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PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements relating to the Testing of Water by the use of Reagents in Tablet Form

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ERRATUM

SPECIFICATION No. 902,884

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Page 2, line 24, after "example" insert
"potassium permanganate or a reducing
agent, for example"

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THE PATENT OFFICE
4th September 1962

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solutions, buffer solutions and/or related appa-
25 ratus and chemicals usually made use of in the
testing of water. Such procedures are not
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chemical techniques and manipulation, because
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We have found that this disadvantage can
be entirely or largely overcome by the
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35 of compressed tablets wherein the required
amounts of the necessary chemical reagents are
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ment and administration to the portion of
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An object of the invention is to provide a
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or substances being determined.

Whereas combinations of indicator and re-
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Miles Laboratories (Br. Patent No. 686,817), 70
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To apply such simplified system of tableted
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COMPLETE SPECIFICATION

Improvements relating to the Testing of Water by the use of Reagents in Tablet Form

We, ARTHUR THOMAS PALIN, a British subject, of "Brentwood", 2, Main Road, Kenton Bank Foot, Newcastle upon Tyne, 3, and WILKINSON AND SIMPSON, LIMITED, a British Company, of Galen House, Low Friar Street, Newcastle upon Tyne, 1, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

In connection with the testing of water in swimming pools or at waterworks and of water used in steam generation it is desirable to know the nature and quantity of dissolved substances in the water including any substance or substances added during the course of treatment, and to know the changes induced in the water by any such treatment.

Methods that are available for the performances of the desired tests require the use of chemical apparatus, standard solutions of acids and alkaline reagents, chemical indicators solutions, buffer solutions and/or related apparatus and chemicals usually made use of in the testing of water. Such procedures are not entirely satisfactory for use by operators unskilled or only partly skilled or untrained in chemical techniques and manipulation, because such lack of skill or training may lead to significant errors in the results obtained.

We have found that this disadvantage can be entirely or largely overcome by the employment of reagents made up in the form of compressed tablets wherein the required amounts of the necessary chemical reagents are compounded together in such a way as to obviate the need for their separate measurement and administration to the portion of water undergoing test.

An object of the invention is to provide a tablet for the testing of a sample of water to

evaluate substances therein, which tablet contains a standard amount of a chemical (hereinafter referred to as a standard reagent) able under conditions of test to react quantitatively with the substance to be determined, together with a quantity of an indicator able by change of colour to denote completion of the quantitative reaction between said standard reagent and said substance, said quantity of indicator being such that the colour imparted thereby to the sample of water is controlled so as to facilitate detection of said change of colour.

A further object of the invention is to provide a method of testing a sample of water for the evaluation of substances therein, which method comprises the steps of taking a predetermined volume of water as said sample and adding thereto, if more than one, in succession, the minimum number of tablets each containing a standard reagent and an indicator as aforesaid necessary to produce a colour change which defines the end point of the reaction between the reagent and the substance or substances being determined.

Whereas combinations of indicator and reagent have been employed in the diagnostic testing of urine and body fluids for instance by Miles Laboratories (Br. Patent No. 686,817), in the testing of water as hereinbefore first referred to, it has previously been the practice where indicators have been used in conjunction with tablets containing a standard reagent to make a separate addition of the indicator either in tablet or solution form. In order to reduce these procedures to their simplest form it is desirable that the standard reagent appropriate to a particular test and the corresponding indicator should be compressed together in the same tablet.

To apply such simplified system of tableted reagents to water testing therefore, it is necessary to determine for each test a suitable

amount of indicator to be incorporated in each tablet in order that the determination of the end points shall be possible by visual detection, and further objects of the invention consist in the provision of various tablets within the generic specification aforesaid which are suitable for the evaluation of the several kinds of substances that are usually found in waters hereinbefore first referred to and which can be evaluated by the stated method or a simple variant thereof.

The invention may, therefore, be said to reside in the incorporation in the same tablet of a specific combination of a standard reagent and an indicator. The reagent-indicator couple may be a polyaminocarboxylic acid, for example ethylene-diamine tetra-acetic acid or a salt thereof, and a metal indicator, for example Eriochrome Black T or Murexide (ammonium purpurate); sulphamic acid or a soluble bisulphate, for example potassium bisulphate, and a pH indicator, for example methyl orange, methyl red, thymol blue or phenolphthalein; an oxidising agent, for example ferrous ammonium sulphate, and an oxidation-reduction indicator, for example methylene blue; a soluble silver salt, for example silver nitrate, and a soluble chromate, for example potassium chromate; sodium carbonate or potassium carbonate and a pH indicator, for example phenolphthalein or thymol blue; a soluble iodate-iodide mixture, for example potassium iodate with potassium iodide, and an iodine indicator, for example starch; and a soluble permanganate, for example potassium permanganate, used in the presence of a soluble iodide and an acidifying agent, for example a soluble bisulphate, with an iodine indicator, for example starch.

Tablets so prepared may be applied to such determinations as the hardness, using for example a combination of disodium ethylenediamine tetra-acetate and Eriochrome Black T, the alkalinity, using for example a combination of potassium bisulphate and methyl red, the dissolved oxygen content, using for example a combination of ferrous ammonium sulphate and methylene blue, the chloride content, using for example a combination of silver nitrate and potassium chromate, the free carbon dioxide content, using for example a combination of sodium carbonate and phenolphthalein, the sulphate content, using for example a combination of disodium ethylenediamine tetra-acetate and Eriochrome Black

T, the sulphite content, using for example a combination of a potassium iodide-potassium iodate mixture and starch, and the content of organic matter as measured by the oxygen absorbed from acid permanganate, using, for example, a combination of potassium permanganate and starch in the presence of potassium iodide and potassium bisulphate.

It will be appreciated that, depending upon the test performed by the method according to the invention, the tablet may also contain ancillary chemicals such as buffering agents and interference-suppressing or masking agents as described for example in "Analytical Applications of Diamino-Ethane-Tetra-Acetic Acid" by T. S. West and A. S. Sykes (published by the British Drug Houses Ltd., England) and in standard reference books such as, for example, "Standard Methods for the Examination of Water, Sewage and Industrial Wastes," Tenth Edition, 1955, American Public Health Association. In addition it may be necessary to incorporate such other substances as are commonly used in the manufacture of compressed tablets to serve as fillers (e.g., sodium chloride, sucrose), disintegrants (e.g., starch) and/or lubricants (e.g., talcum, boric acid).

The nature of the tablets provided by our invention, and of the method of testing water further provided thereby, will be understood from the following selected but not exhaustive Examples, forming part of this specification, of the compositions of tablets, and description of modes of carrying out tests, for evaluating various substances in samples of water (whether or not diluted with distilled, i.e., pure water) having volumes usually of 50 ml, 100 ml or 200 ml.

EXAMPLE I.

Total Hardness Test.

In determining the substances which give rise to the hardness of water we use as the standard reagent a polyaminocarboxylic acid, such as ethylene-diamine tetra-acetic acid, or a salt thereof, in conjunction with a metal indicator such as Eriochrome Black T. When using Eriochrome Black T we find that a suitable amount to incorporate in each tablet is 0.5 mg which may be increased in a tablet for testing boiler waters to 2.0 mg. In conformity with this discovery, a suitable mixture for the preparation of the hardness test tablets may be formulated as follows:—

Reagent	Ethylene-diamine tetra-acetic acid (Disodium salt)	7.5 g
Indicator	Eriochrome Black T	0.5 g
Buffer	Sodium borate	16.0 g
	Sodium hydroxide	4.0 g
Interference Suppressor	Sodium diethyl dithiocarbamate	2.0 g
Filler	Sodium Chloride	50.0 g
Disintegrant	Starch	10.0 g
Lubricant	Talcum	10.0 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.1 g weight.

from such modified mixture must be of a proportionately increased weight.

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EXAMPLE IA.

Total Hardness Boiler Water Test.

When the tablets are to be used for testing boiler waters, the same formulation may be followed except that the amount of Eriochrome Black T may be increased to 2.0 g and the amount of sodium chloride reduced to 48.5 g. With boiler water samples it may also be advantageous to increase the amount of the buffering agents, sodium borate and sodium hydroxide, in which case the tablet prepared

EXAMPLE II.

Calcium Hardness Test

When it is required to test only for the calcium salts causing hardness, we employ a metal indicator such as Murexide (ammonium purpurate) and we find that a suitable amount to incorporate in each tablet is 0.2 mg. In conformity with this discovery, a suitable mixture for the preparation of the calcium hardness test tablets may be formulated as follows:—

Reagent	Ethylene-diamine tetra-acetic acid (disodium salt)	7.5 g
Indicator	Murexide	0.2 g
Filler	Sodium Chloride	72.3 g
Disintegrant	Starch	10.0 g
Lubricant	Talcum	10.0 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.1 g weight.

EXAMPLE III.

Alkalinity to Methyl Orange Test.

In determining the substances which give rise to the "Alkalinity to methyl orange" of water (i.e., Alkalinity M) we use as the standard reagent an acidic substance such as a bisulphate in conjunction with methyl orange,

methyl red or other indicator, exhibiting a colour change over a pH range within the limits 2.9 to 6.3. When using methyl red (water soluble) which because of its greater solubility is preferable to methyl orange we find that a suitable amount to incorporate in each tablet is 0.4 mg in conformity with which discovery a suitable mixture for the preparation of the Alkalinity M test tablets may be formulated as follows:—

Reagent	Potassium bisulphate	11.0 g
Indicator	Methyl red (water soluble)	0.4 g
Filler	Sodium Chloride	68.6 g
Disintegrant	Starch	10.0 g
Lubricant	Talcum	10.0 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.1 g weight.

EXAMPLE IIIA.

Low Alkalinity to Methyl Orange Test

With waters of low alkalinity M it may be advantageous to use half strength tablets wherein the amount of potassium bisulphate is reduced to 5.5 g and the amount of sodium chloride increased to 74.1 g.

EXAMPLE IV.

Alkalinity to phenolphthalein Test.

In determining the substances which give

rise to the "Alkalinity to phenolphthalein" of water (i.e., Alkalinity P) we use as the standard reagent an acidic substance such as a bisulphate in conjunction with phenolphthalein, thymol blue or other indicator exhibiting a colour change over a pH range within the limits 8.0 to 10.0. When using thymol blue which because of its greater solubility is preferable to phenolphthalein we find that a suitable amount to incorporate in each tablet is 0.4 mg in conformity with which discovery a suitable mixture for the preparation of the Alkalinity P test tablets may be formulated as follows:—

Reagent	Potassium bisulphate	11.0 g
Indicator	Thymol Blue	0.4 g
Filler	Sodium Chloride	68.6 g
Disintegrant	Starch	10.0 g
Lubricant	Talcum	10.0 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.1 g weight.

EXAMPLE IVA.

Low Alkalinity P.

With water of low alkalinity P it may be advantageous to use half strength tablets wherein the amount of potassium bisulphate is reduced to 5.5 g and the amount of sodium chloride increased to 74.1 g.

EXAMPLE V.

Chloride Test.

In determining the chloride content of water we use as the standard reagent a soluble silver salt such as silver nitrate in conjunction with an indicator consisting of a chromate such as potassium chromate. When using potassium chromate we find that a suitable amount to incorporate in each tablet is 20 mg in conformity with which discovery a suitable mixture for the preparation of the Chloride Test tablets may be formulated as follows:—

Reagent	Silver Nitrate	4.8 g
Indicator	Potassium chromate	20.0 g
Filler	Potassium bicarbonate	55.2 g
Disintegrant	Starch	10.0 g
Lubricant	Talcum	10.0 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.1 g weight.

EXAMPLE VI.

Sulphate Test.

In determining the sulphate content of water we first add to the sample a suitable amount of an acid and, at the same time or following a known excess of barium chloride either in solution or tablet form. By this means any sulphates present in the water are precipitated as barium sulphate. The Sulphate Test tablets are then used to determine the amount of barium chloride remaining in solution in the water so providing an indirect determination

of the sulphate content of the original sample. For the purpose of this determination we use as the standard reagent an aminopolycarboxylic acid, such as ethylene-diamine tetra-acetic acid, or a salt thereof, in conjunction with a metal indicator such as Eriochrome Black T. When using Eriochrome Black T we find that a suitable amount to incorporate in each tablet is 0.5 mg. In conformity with this discovery a suitable mixture for the preparation of Sulphate Test Tablets (which may be suitably identified, for example, as Sulphate Test Tablets No. 2 in the event that the barium chloride first added is prepared in tablet form) may be formulated as follows:—

Reagent	Ethylene diamine tetra-acetic acid (disodium salt)	2.63 g
Indicator	Eriochrome Black T	0.50 g
Buffers	Sodium borate	34.00 g
	Sodium hydroxide	10.00 g
Filler	Sodium Chloride	112.87 g
Disintegrant	Starch Powder	20.00 g
Lubricant	Talcum	20.00 g

The dry mixture is passed through a B.S.36 mesh sieve and compressed into tablets of 0.2 g weight.

It may be desirable to increase the amount of sodium hydroxide in the above mixture should there be any difficulty when using the tablets in raising the pH of the acidified sample to a value of 10 or thereabouts.

In the application of the method of testing with a selected one of the tablets according to the aforesaid example, it is in general required only to add, if more than one, in succession, to a sample of water of suitable volume the minimum number of tablets required to cause a colour change of the indicator and depending upon the make-up of the tablets the substance content of the water is calculable within the range given by the tablet.

Thus, in determining the total hardness of a raw water or a treated water (as in a water works or a swimming bath) it is required only to add to a sample of 100 ml of the water in a stoppered bottle a sufficient number of the tablets according to Example I, with shaking, until the red tinge caused by the indicator disappears then to multiply the number of tablets used by 20 (since each tablet is the equivalent of 20 ppm impurities) and deduct 10, i.e., the equivalent of half a tablet, to correct for overshooting. This gives an

evaluation with an average accuracy of +5 ppm according to the formula, for a 100 ml sample:—

$$(1) \text{ Hardness} = (\text{No. of tablets I} \times 20) - 10 \text{ ppm.}$$

If a sample of different volume is taken, the formula is correspondingly varied, e.g., for a 50 ml sample the formula is:—

$$(2) \text{ Hardness} = (\text{No. of tablets I} \times 40) - 20 \text{ ppm.}$$

For determining the low hardness of waters such as boiler waters, softened waters and condensates, the tablet according to Example IA is used, each of which tablets is the equivalent in a 100 ml sample of 2 ppm. Thus the formula for evaluating the hardness of such a water is:—

$$(3) \text{ Hardness} = (\text{No. of tablets IA} \times 2) - 1 \text{ ppm.}$$

It is to be noted that the tablets according to Examples I and IA include buffering agents and an interference suppressor. For the testing of boiler waters of high caustic alkalinity, if the tablets do not include the higher amounts of buffers as stated in Example IA, the method

may be modified by the prior addition to the sample of supplementary buffer tablets to adjust the pH value to 10 or thereabouts, such tablets of course not being counted in the calculation according to the formula (3) above.

When testing raw or treated waters for calcium hardness only, before adding to the sample of water being tested a number of tablets according to Example II above, it is necessary that the pH value of the water be raised by the addition of an alkali such as sodium hydroxide, which may also be made available in the form of pellets. These are of course not counted in the determination of the calcium hardness, according to the volume of the sample, by the formula (1) or (2) above stated, substituting therein the number of tablets according to Example II which cause a change of colour from pink to violet.

A similar method is employed for testing alkalinity with tablets according to either Example III or Example IV, each of which is equivalent to 20 ppm in a sample of 200 ml or to 40 ppm in a sample of 100 ml; or alternatively with half strength tablets according to either Example IIIA or Example IVA, which are equivalent to 10 ppm in a sample of 200 ml or 20 ppm in a sample of 100 ml. In the appropriate formula there is substituted the number of tablets III or IIIA required to change the colour from yellow to bright pink, or the number of tablets IV or IVA required to change the colour from blue to yellow.

When it is desired to test the alkalinity of boiler waters in the presence of barium chloride, there may first be added to the sample barium chloride in tablet form, whereafter the test is carried out and evaluated with the use of tablets according to Example IV or Example IVA as above described, according to the expected degree of alkalinity.

There are no special requirements for testing for chloride with the aid of tablets according to the foregoing Example V (wherein completion of the reaction is indicated by a colour change from yellow to reddish brown) except that it is desirable to use small sample volumes for boiler waters and high sample volumes for condensates. The tablet according to Example V contains the equivalent of 5 ppm in a volume of 200 ml water, or 100 ppm in a volume of 10 ml water, with intermediate values in proportion.

In any of the foregoing methods of testing, for results having an accuracy equal to or approaching that of the corresponding laboratory methods, the following "back-titration" procedure may be used:—

Measure 100 ml of sample in a graduated jar, but pour only about $\frac{2}{3}$ of it into the graduated stoppered bottle and with shaking add the tablets in the usual way until the required colour change is obtained. At this stage gradually pour the remainder of the sample into the bottle a little at a time, shaking

until the colour just begins to turn back again. Note how much sample is left unused in the graduated jar. The result is then given by the following formula:—

$$\frac{\text{No. of Tablets used} \times T \times 100}{(100 - V)} \quad 70$$

where "T" is the tablet value in terms of the impurities sought and "V" is the volume left in the graduated jar.

The method of testing for sulphate with tablets according to Example VI is set out in that example, since it is necessary to precipitate the sulphate with barium chloride, preferably in the tablet form, and thereafter use tablets of the specified formula to estimate residual barium chloride. The barium chloride added may be made equivalent to 2800 ppm of sulphate, and the test tablets are equivalent each to 200 ppm in a sample of 5 ml and the impurity is evaluated according to the formula:—

$$(4) \text{ Sulphate} = 2800 - (\text{No. of tablets VI} \times 200).$$

If the sulphate can be assumed to be more than 2000 ppm Na_2SO_4 or is determined to be so by a preliminary test, it is desirable to use a sample of 2 ml volume and then multiply the result of formula (4) by 2.5.

WHAT WE CLAIM IS:—

1. A tablet for the testing of a sample of water to evaluate substances therein by the use of a combination of a standard reagent and an indicator as hereinbefore defined the said reagent and indicator being compressed together in the same tablet.

2. A tablet according to Claim 1 which contains an amount of indicator such that the colour change is observable visually when the necessary number of tablets have been added for the quantitative reaction between the standard reagent and the substance or substances to be completed.

3. A tablet according to Claim 1 or Claim 2 which also contains at least one of buffering and masking agents which act to suppress interference from foreign ions in determining the said substance or substances.

4. A tablet according to any of Claims 1 to 3 which also contains at least one of filler, disintegrant and lubricant substances to enable the production of the tablet in stable form while dry.

5. A tablet according to Claim 1 substantially as hereinbefore described with reference to the examples.

6. A method of testing a sample of water for the evaluation of a substance or substances therein which method comprises the steps of taking a predetermined volume of the water as said sample and adding thereto, if more than

one, in succession, the minimum number of tablets constituted in accordance with any of Claims 1 to 5 necessary to produce a colour change which defines the end point of the reaction between the reagent and the substance or substances being determined and permits the substance content to be calculated by reference

to said minimum number.

7. A method of testing water according to Claim 6 substantially as hereinbefore described with reference to the examples.

A. T. PALIN,

W. H. THOMAS,

For Wilkinson & Simpson, Ltd.

PROVISIONAL SPECIFICATION

Improvements relating to the Testing of Water by the use of Reagents in Tablet Form

We, ARTHUR THOMAS PALIN, of Brentwood, 2, Main Road, Kenton Bank Foot, Newcastle upon Tyne, 3, a British Subject, and WILKINSON & SIMPSON, LIMITED, of Galen House, Low Friar Street, Newcastle upon Tyne, 1, a British Company, do hereby declare this invention to be described in the following statement:—

In connection with the testing of water in swimming pools, at waterworks and of water used in steam generation it is desirable to know the nature and quantity of dissolved substances in the water including any substance or substances added during the course of treatment and to know the changes induced in the water by any such treatment.

Methods that are available for the performance of the desired tests require the use of chemical apparatus, standard solutions of acids and alkaline reagents, chemical indicator solutions, buffer solutions and/or related apparatus and chemicals usually made use of in the testing of water. Such procedures are not entirely satisfactory for use by operators unskilled or only partly skilled or untrained in chemical techniques and manipulation because such lack of skill or training may lead to significant errors in the results obtained.

We have found that this disadvantage can be entirely or largely overcome by the employment of reagents made up in the form of compressed tablets wherein the required amounts of the necessary chemical reagents are compounded together in such a way as to obviate the need for their separate measurement and administration to the portion of water undergoing test.

The composition of the tablets must be such as to include a standard amount of a chemical (hereinafter called the standard reagent) able under the conditions of the test to react quantitatively with the substance or substances being determined. From the number of tablets used to complete such quantitative reaction in a given volume of water an estimate may be obtained of the concentration of the dissolved substance or substances sought. In order to provide a visual indication of the completion of the reaction it is necessary that there should also be added to the sample of water a quantity of an indicator which by a change of colour is able sharply to define the end point of the reaction.

Where such indicators have been used in conjunction with tablets containing a standard reagent it has previously been the practice to make a separate addition of the indicator either in tablet or solution form. In order to reduce these procedures to their simplest form, however, it is desirable that the standard reagent appropriate to a particular test and the corresponding indicator should be compressed together in the same tablet. A disadvantage of such a combination, however, is that as more and more of the tablets are added to the sample of water in order to bring the concentration of the standard reagents in the water up to that required for the completion of the quantitative reaction involved in the particular test there is, at the same time, a *pro rata* increase in the concentration of the indicator in the water. Consequently if the total number of tablets applied to a given volume of water is large the colour imparted to the mixture by the amount of indicator present may be so intense as to make it difficult or impossible to judge with the required precision at what stage that change occurs, which serves to denote completion of the particular quantitative reaction concerned. On the other hand, if the amount of indicator contained in each tablet is decreased to avoid imparting too great an intensity of colour in the circumstances above described it may then be found in tests requiring the addition of only a few tablets to the given volume of water that the intensity of the colour thus imparted to the water may be so weak as to provide an insufficiently clear indication of the end point of the determination.

To apply such simplified system of tableted reagents to water testing, therefore, it is necessary to determine for each test a suitable amount of indicator to be incorporated in each tablet in order that the determination of the end points shall be possible by visual observation.

The invention may therefore, be said to reside in a method of incorporating in the same tablet a standard reagent and an indicator, the amount of indicator being such as to permit the application of the tablets to the testing of water. Tablets so prepared may be applied to such determinations as the hardness, alkalinity, chloride and sulphate contents of water.

- For the purpose of these tests, having regard to the requirements from the point of view of simplicity and convenience, twenty tablets are taken as the maximum number that should be used in a single test; one tablet may be taken to represent the minimum number. That being the case while a suitable amount of indicator to be incorporated in the tablet will depend upon such other factors as the nature of the indicator itself and the volume of sample used in the test the essential requirement is that the colour imparted to the sample where the maximum number of tablets is added should not be so intense, nor the colour imparted where the minimum number is added so faint that, in either case, detection of the colour change corresponding to completion of the particular quantitative reaction involved in the test would become difficult or impossible.
- It will be appreciated that, depending upon the test to which the invention is applied, the tablet may also contain ancillary chemicals such as buffering agents and interference-suppressing or masking agents as described in "Analytical Applications of Diamino-Ethane-Tetra-Acetic Acid" by T. S. West and A. S. Sykes (published by The British Drug Houses Ltd.) and in standard reference books such as, for example, "Standard Methods for the Examination of Water, Sewage and Industrial Wastes," Tenth Edition, 1955, American Public Health Association. In addition it may be necessary to incorporate such other substances as are commonly used in the manufacture of compressed tablets to serve as fillers (e.g., sodium chloride, sucrose), disintegrants (e.g., starch), and/or lubricants (e.g., talcum, boric acid).
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